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Fabrication and Characterization of Barium Strontium Titanate and Non-Ferroelectric Oxide Composites

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ARL-TR-657

December 1994



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*Form Approved
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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
		December 1994	
4. TITLE AND SUBTITLE Fabrication and Characterization of Barium Strontium Titanate and Non-Ferroelectric Oxide Composites			5. FUNDING NUMBERS
6. AUTHOR(S) E. Ngo, S. Stowell, L.C. Sengupta, M.E. O'Day, and R. Lancto			
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Army Research Laboratory Watertown, MA 02172-0001 AMSRL-MA-CA			8. PERFORMING ORGANIZATION REPORT NUMBER ARL-TR-657
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)			10. SPONSORING/MONITORING AGENCY REPORT NUMBER
11. SUPPLEMENTARY NOTES			
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited.			12b. DISTRIBUTION CODE
13. ABSTRACT (Maximum 200 words) Various composites of BSTO combined with other nonelectrically active oxide ceramics have been formulated. In general, the composites have adjustable electronic properties which can be tailored for use in various electronic devices. Two applications which are of interest in regard to the composites being discussed here, are phased array antennas and capacitor-varistor monolithic devices. For the phased array antennas, these properties include reduced dielectric constants, ϵ' , where $\epsilon = \epsilon' - i\epsilon''$, and loss tangents, $\tan \delta$. The reduction of the dielectric constant and lowering of the loss tangents reduce the overall impedance mismatch and the insertion loss of the device. In addition, the overall tunability, change in the dielectric constant with applied voltage, is maintained at a sufficiently high level for all dielectric constants of interest. Another application (for one of the composite materials) is a combined capacitor-varistor device. In general, this would be used to protect against spurious voltage surges and voltage transients in electronic micro-circuits. The microstructures including grain size and phase analysis have been examined using SEM and X-ray diffraction.			
14. SUBJECT TERMS BSTO, Phased Array Antennas, Composites			15. NUMBER OF PAGES 9
			16. PRICE CODE
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL

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INTRODUCTION

Phased array antennas can steer transmitted or received signals either linearly or in two dimensions without mechanically oscillating the antenna. These antennas are currently constructed using ferrite phase shifting elements. Due to the type of circuit requirements necessary to operate these antennas, they are costly, large and heavy. Therefore, the use of these antennas has been limited primarily to military applications which are strategically dependent on such capabilities. In order to make these devices available for many other commercial and military uses, the basic concept of the antenna must be improved. If ferroelectric materials could be used for the phase shifting element instead of ferrites, phased array antennas would be totally revolutionized. A ceramic Barium Strontium Titanate, $Ba_{1-x}Sr_xTiO_3$, (BSTO), phase shifter using a planar microstrip construction has been demonstrated.¹ In order to meet the required performance specifications, maximum phase shifting ability, the electronic properties must be optimized. As part of this optimization process, various composites of BSTO and non-ferroelectric oxides have been formulated.

Another application for one of these materials is a combined capacitor-varistor device. In general, this would be used as a protective device in parallel with electronic information processing circuits to protect against spurious voltage surges and voltage transients. The capacitive aspect of the device would guard against low-amplitude and high frequency transients which cause errors in signal processing or in stored signals. The varistor function of the device protects against high-amplitude voltage surges. The capacitor-varistor device has been attempted previously by appropriately doping $SrTiO_3$ to form grain boundary phases or layers which exhibit the desirable characteristics.² Also discrete layers of ZnO and $Pb(Fe_{1/2}Nb_{1/2})O_3$ - $Pb(Fe_{2/3}W_{1/3})O_3$ (dielectric constant of 27000 to 32000) have been fabricated.³ The problem with the former scheme has been with the high loss tangents and high threshold voltages derived from the metallic additives and the semiconducting capacitor. The problem associated with the latter scheme is with the processing of the lead based capacitor compounds and the deleterious diffusion between the ZnO varistor layers and lead-based capacitor layers.

The composites will be designated as BSTO-Oxide II, BSTO-Oxide III, BSTO-Oxide II / BSTO-Oxide III, BSTO-Oxide III Compound (A), BSTO-Oxide III Compound (B), BSTO-Oxide III Compound (C), and BSTO-Oxide IV since they all have patents pending on their formulations. All of these composites possess improved electronic properties. The comparison of the compositions and phase formation of the various BSTO-Oxide ceramic composites will be made and related to their electronic properties.

EXPERIMENTAL

Processing and Metallization

The ceramics have been processed using standard solid state methods and which have been outlined in a previous publication.⁴ The electrodes were fabricated using two metallization techniques. One involved painting on two circular, aligned electrodes, one on either side of the specimens, using high purity silver paint (SPI Supplies West Chester, PA) and attaching wires using high purity silver epoxy, Magnobond 8000, made by Magnolia Plastics, Inc., Chamblee, GA. The other technique utilized the screen printing of electrodes using silver conductive ink

(FERRO #3350, Electronic Materials Division, Santa Barbara, CA) and wires were attached by dipping the specimens in a bath of 2% silver, 62% tin and 36% lead solder.

Electronic Measurements

The dielectric constants, ϵ' , loss, $\tan \delta$, and % tunability and Curie temperatures were determined for all composites. The % tunability of a material is determined using the following equation:

$$\% \text{ tunability} = \{ \epsilon'(0) - \epsilon'(\text{Vapp}) \} / \{ \epsilon'(0) \} \quad (1)$$

The tunability measurements were taken with an applied electric field which ranged from 0 to 3.0 V/micron (μm). The electronic properties given in the forthcoming tables were measured at a frequency of 1 KHz. Capacitance measurements for all materials were taken using an HP4284A LCR meter. Further calculations were done to correct for the effect of fringe capacitance.

RESULTS AND DISCUSSION

BSTO-Oxide III, BSTO-Oxide II / Oxide III, BSTO-Oxide III Compound Composites

SEM and X-ray Diffraction: No secondary phases (other than BSTO and the Oxide III components) were identified by X-ray diffraction for the BSTO-Oxide III and BSTO-Oxide III Compound (B) composites. Also the SEM photographs did not reveal any evidence of secondary phase formation. However, analysis of the X-ray diffraction patterns of the BSTO-Oxide II/Oxide III, BSTO-Oxide III Compound (A) and BSTO-Oxide III Compound (C) composites revealed the existence of secondary phases. These phases were also evident as discolorations in their respective SEM photographs.

Electronic Properties: The electronic data for the BSTO-Oxide III, BSTO-Oxide II / Oxide III and BSTO-Oxide III Compound (A)-(C) composites are shown in Table 1. As shown in the table, the dielectric constants decrease with increase in oxide content and the tunability decreases slowly with increase in oxide content. In fact, the BSTO-Oxide III and BSTO-Oxide III Compound (B) composites exhibit high tunabilities (>10%) up to 60 wt% oxide III which is not the case for the other composites reported in the table. However, at similar electric field strengths the tunability of the 60 wt% BSTO-Oxide III Compound (B) composite is nearly twice that of the 60 wt% BSTO-Oxide III composite. This may be due to the fact that, the Curie temperature of the 60 wt% BSTO-Oxide III Compound (B) composite is at 0°C . On the other hand, the Curie temperatures for the 60 wt% BSTO-Oxide III, BSTO-Oxide II/Oxide III and the BSTO-Oxide III Compound (A) and BSTO-Oxide III Compound (C) composites are all very low, <-50 to <-55 $^{\circ}\text{C}$, (in the far *paraelectric* region). Another significant factor which effects the tunability is the size of the oxide additive. The size of Oxide II is much greater than the size of Oxide III. Therefore composites containing Oxide II, BSTO-Oxide II / Oxide III and BSTO-Oxide III Compound (A) (which also contains Oxide II), will have much less tunability at high additive content (60 wt%) due to lack of connectivity between the BSTO in the composites. The observed tunabilities for these materials is <2.0%. The differences in the tunabilities of the 60 wt% oxide content composites could also be due to the fact that the BSTO-Oxide III and BSTO-

TABLE I: Electronic Properties of BSTO-Oxide III, BSTO-Oxide II / Oxide III, BSTO-Oxide III Compound (A), BSTO-Oxide III Compound (B), and BSTO-Oxide III Compound (C) Ceramic Composites Measured at 1 KHz. (*poor contact)

BSTO-Oxide III

Oxide III Content (wt%)	Dielectric Constant	Loss Tangent	% Tunability	Electric Field (V/ μ m)	Curie Temp (C)
0.0	3299.08	0.0195	19.91	0.73	10
1.0	1276.21	0.0015	16.07	2.32	-30
5.0	1770.42	0.0014			-30
10.0	1509.19	0.0018			-35
20.0	1079.21	0.0009	15.95	2.33	-30
30.0	750.93	0.0008	9.350	1.62	-30
60.0	117.67	0.0006	11.08	2.70	<-55

BSTO-Oxide II / Oxide III

Oxide II / Oxide III Content (wt%)	Dielectric Constant	Loss Tangent	% Tunability	Electric Field (V/ μ m)	Curie Temp (C)
0.0	3299.1	0.0195	19.91	0.73	10
1.0	2515.3	0.0011	12.24	1.14	-15
10.0	1868.9	0.0013	11.63	1.56	-20
20.0	1016.0	0.0327*	10.89	1.33	<-55
30.0	389.06	0.0009	1.607	1.34	<-55
60.0	93.591	0.0022	0.450	0.99	<-55

BSTO-Oxide III Compound (A)

Oxide III Compound (A) Content (wt%)	Dielectric Constant	Loss Tangent	% Tunability	Electric Field (V/ μ m)	Curie Temp (C)
0.0	3299.1	0.0195	19.91	0.73	10
1.0	3064.9	0.0019	18.58	1.66	-5
10.0	2209.2	0.0019	13.29	1.34	-5
20.0	1425.7	0.0014	6.910	0.91	-10
30.0	582.84	0.0030	7.347	0.51	-30
60.0	83.941	0.0176*	1.054	1.00	<-50

BSTO-Oxide III Compound (B)

Oxide III Compound (B) Content (wt%)	Dielectric Constant	Loss Tangent	% Tunability	Electric Field (V/ μ m)	Curie Temp (C)
0.0	3299.1	0.0195	19.91	0.73	10
1.0	3292.5	0.0066	35.24	2.00	-10
5.0	2267.4	0.0050	29.78	2.00	0
10.0	1264.6	0.0057	19.94	2.00	-25
20.0	842.23	0.0146	19.93	2.00	-20
30.0	413.04	0.0313	22.72	2.00	-10
60.0	76.70	0.0125	17.00	2.00	0

BSTO-Oxide III Compound (C)

Oxide III Compound (C) Content (wt%)	Dielectric Constant	Loss Tangent	% Tunability	Electric Field (V/ μ m)	Curie Temp (C)
0.0	3299.1	0.0195	19.91	0.73	10
1.0	2256.5	0.0014	30.96	2.00	-17
5.0	2193.2	0.0021	17.89	2.00	-15
10.0	1386.5	0.0022	18.69	2.00	-15
20.0	649.90	0.0021	15.23	2.00	-13
30.0	263.52	0.0056	11.52	2.00	-50
60.0	108.25	0.0085	3.72	2.00	<-55

Oxide III Compound (B) compounds do not exhibit secondary phases whereas the other composites form multiple secondary phases as discussed previously. These non-ferroelectric phases tend to inhibit tunability at high additive contents.

As shown in Fig. 1, the loss tangents of most of these composites are extremely low (< 0.01). However, the loss tangents of the BSTO-Oxide III Compound (B) composites are greater than 0.01 and all of the other composites reported here. Again, the position of the Curie temperature for these composites may contribute to the higher losses evident in these specimens. In fact, the Curie temperature and the loss of these composites is much closer to that of undoped BSTO ($\text{Ba}=0.60$).

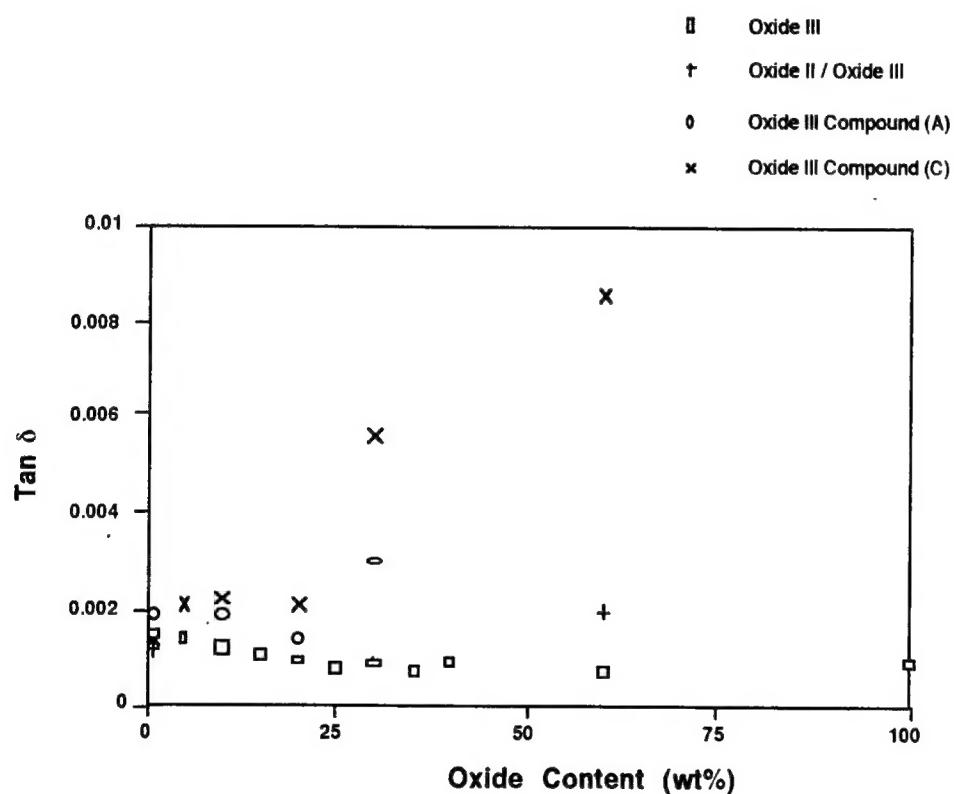


Fig. 1. Loss Tangent vs. Oxide Content for BSTO-Oxide III, BSTO-Oxide II/ Oxide III and BSTO-Oxide III (A) and BSTO-Oxide III (C) Compound Composites measured at 1 KHz.

BSTO-Oxide IV Composites

The electronic properties and the average grain size of the BSTO-Oxide IV composites are shown in Table 2. At low doping levels (1-15 wt%), the composites have large dielectric constants and possess low loss tangents and reasonably high tunabilities. These properties meet the requirements for various antenna applications (especially low frequency applications). At higher levels (20-50 wt%), the addition of oxide IV content continues to increase the dielectric constant drastically especially at low frequencies, and the composites begin to conduct current and exhibit non-ohmic behavior and the threshold voltage is seen to increase with an increase in oxide IV content. The nonlinear electrical conduction in the ceramic composite is due to the addition of oxide IV (no secondary phases were observed from X-ray diffraction and SEM analysis) in which tunneling through the grain boundaries occurs.

Fig. 2 shows the current versus voltage for BSTO-Oxide IV (30 and 50 wt%) composites. It should be noted that as the oxide IV content is increased, the dielectric constant increases, and the non-linearity exponent increases. This increase in the non-linearity is also roughly related to the decrease in the grain size as indicated in Table 2.

The threshold voltage for the BSTO-Oxide IV (50 wt%) specimen is lower than reported for any bulk capacitor-varistor device (which was reported to be around 100 V)² and the capacitance is higher than that obtained for multilayered structures (nearly twice the capacitance reported)³.

TABLE 2. Electronic Properties and Grain Size of BSTO-Oxide IV Composites
Measured at 1 KHz.

<u>Oxide IV Content wt.%</u>	<u>Dielectric Constant</u>	<u>Loss Tangent</u>	<u>Tunability (Percent)</u>	<u>Electric Field (V/μm)</u>
1.0	3756	0.00236	7.334	1.0
5.0	3416	0.01276	8.957	0.8
10.0	3908	0.01320	13.11	0.7
15.0	3942	0.03708	27.97	0.6
20.0	4685	0.19113	—	—
25.0	7520	0.46976	—	—
30.0	7859	0.46927	—	—
50.0	71922	0.46891	—	—
<u>Oxide IV Content wt.%</u>	<u>Threshold Voltage</u>	<u>Nonlinearity Exponent</u>	<u>Grain Size (μm)</u>	
1.0	—	—	—	—
5.0	—	—	10.24	
10.0	—	—	9.774	
15.0	—	—	8.610	
20.0	100	6.510	8.387	
25.0	25	5.390	7.514	
30.0	20	10.290	6.806	
50.0	5	8.349	5.909	

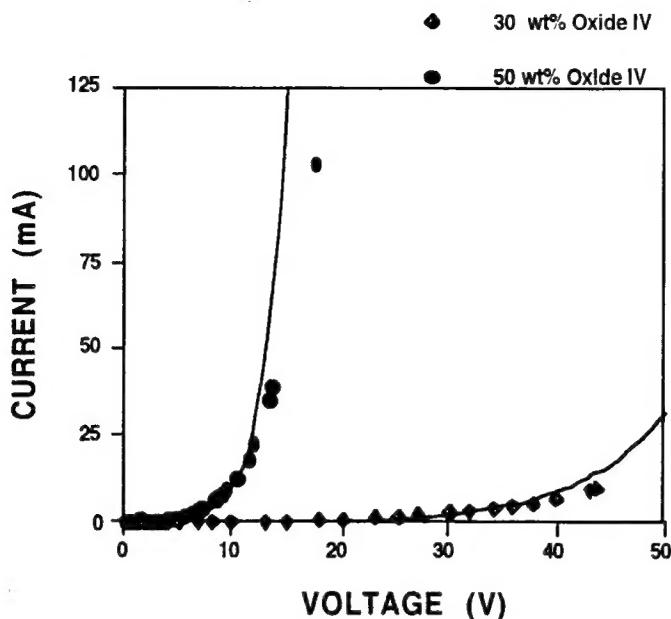


Fig. 2. Current vs. Voltage for BSTO- Oxide IV (30 and 50 wt%) Composites.

CONCLUSIONS

The BSTO-Oxide III, BSTO-Oxide II / Oxide III and BSTO-Oxide III Compound (A)-(C) ceramic composites possess low loss tangents and high tunabilities. The data suggest that composites containing oxide III or an oxide III compound tend to display similar electronic behavior and can potentially facilitate the operation of ferroelectric phased array antennas at millimeter wave range frequencies. The BSTO-Oxide IV materials exhibit behavior which renders them suitable for use both (at oxide content < 15 wt%) in phased array antenna systems and (at oxide content > 15 wt%) as a capacitor-varistor protection device for microelectronic circuits. The improved properties of these composites includes very low threshold voltages (< 5 V), reasonable nonlinear coefficients, moderate to low loss tangents, tunable and extremely high dielectric constants. It should be noted that the low threshold voltage accompanied by an extremely high dielectric constant was accomplished in a bulk ceramic (1mm thickness) and is less than that reported for thin film laminates³.

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